Federal Democratic
Republic of Ethiopia

☞ EDICT OF GOVERNMENT ☜

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ET 310 (2001) (English): EDIBLE SALT - DETERMINATION OF IRON CONTENT - 1, 10-phenanthroline spectrophotometric method

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ETHIOPIAN STANDARD

ES 310:2001

(Reaffirmed:2012)

Second edition

Edible salt - Determination of iron content -1,10-phenanthroline spectrophotometric method

ICS: 67.220.20

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Foreword

This Ethiopian Standard has been prepared under the direction of the Technical Committee for Food additives and contaminants (TC 21) and published by the Ethiopian Standards Agency (ESA).

The standard is a reaffirmation for reprint of the Ethiopian Standard ES 310:2001, Edible salt – Determination of iron content -1,10-phenanthroline spectrophotometric method, with some editorial changes without altering the technical contents in the former text.

Edible salt — Determination of iron content — 1,10-phenanthroline spectrophotometric method

1 Scope

This Ethiopian Standard specifies the method for the determination of Iron content in salt.

2 Normative references

The following standard contain provisions which through reference in this text, constitute the provisions of this Ethiopian Standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreements based on this Ethiopian Standard are encouraged to investigate the possibility of applying the most recent editions of the Ethiopian Standards indicated below. Registers of currently valid standards are maintained in the Quality and Standards Authority of Ethiopia.

ES 299:2001, *Edible salt - Sampling.*

ES ISO 2483:2001,*Sodium Chloride for Industrial Use – Determination of the loss of mass at 110 °C.*

3 Principle

a) The reduction of trivalent iron by hydroxylammonium chloride; and the formation of a coloured complex between divalent iron and 1, 10- Phenanthroline in buffered medium;

b) The photometric measurement of the coloured complex at wavelength of approximately 510 nm.

4. Reagents

The following reagents of recognized analytical reagent quality shall be used:

4.1 Hydrochloric acid, 1.19 g/ml, 37% (m/m)

4.2 Acetic acid glacial, 1.05 g/ml, 99% to 100% (m/m) solution

4.3 Hydroxylammonium chloride solution - Dissolve 10 g of hydroxylammonium chloride ($NH_2OH.HCl$) in 1L of water

4.4 1,10-phenanthroline solution - Dissolve 2.5 g of 1,10 phenanthroline hydrochloride monohydrate ($C_{12}H_8N_2$. $HCl.H_2O$) in 1 L of water.

NOTE. - This compound may be replaced by 2.1g 1,10 -phenanthroline monohydrate ($C_{12}H_8N_2H_2O$).

4.5 Sodium acetate solution - Dissolve 500 g sodium acetate trihydrate ($CH_3COONa.3H_2O$) in water and dilute to 1 L.

4.6 Buffer solution - Dissolve 272 g of sodium acetate trihydrate ($CH_3COONa.3H_2O$) in approximately 500 ml of water. Add 240 ml of the acetic acid to the solution. Transfer quantitatively to 1000 ml one mark volumetric flask, dilute to the mark with water and mix well. This solution has a pH of 4.9.

4.7 Iron stock solution, corresponding to 0.010g/L Fe - Transfer 25 ml of the standard iron solution to a mark 500 ml one mark volumetric flask, dilute to the mark with water and mix well.1ml of this solution contains 10 µg of Fe. Prepare this solution at the time of use.

5. Apparatus

5.1 Spectrometer, or photometer, fitted with filters, which provide a transmission maximum between 500 nm and 520 nm.

5.2 pH meter equipped with a glass electrode and a calomel reference electrode, or pH indicator paper, covering the pH range 3.5 to 4.2 and indicating in 0.2 pH unit intervals.

6. Sampling

Samples shall be taken in accordance with ES 299.

7. Procedure

7.1 Caution

It shall be ensured that no trace of iron is introduced during the analysis by the reagents, the water or the apparatus especially used in the preparation of the initial sample.

7.2 General

All new glasswares used in the determination shall be washed as follows:

a) with brush and detergent if the sides are greasy;

b) with concentrated nitric acid, followed by several rinses with water.

7.3 Test portion

Weigh, to the nearest 0.01g, 10g of the test sample.

7.4 Blank test

Blank test shall be carried out following the same procedure and using the same quantities of all the reagents used for the determination but omitting the test portion.

7.5 Preparation of the calibration curve

7.5.1 Preparation of the standard calorimetric solutions

The volumes of the standard calorimetric solutions

The volumes of the standard iron solution given in table 1 shall be added to a series of five 100 ml one mark volumetric flasks.

The solution shall be diluted in each flask to approximately 50 ml with water, and then add the following:

a) 0.5 ml of the hydrochloric acid solution;

b) 5 ml of the hydroxylammonium chloride solution;

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c) 5 ml of the 1, 10-phenantroline solution;

d) 25 ml of the buffer solution - Dilute to the mark with water, mix well and allow the solution to stand for 10 min.

Table 1 – Mass of iron in standard solutions

Standard iron solution	Corresponding mass of iron
ml	µg
0[1]	—
2.5	25
5.0	50
10.0	100
15.0	150

[1] Compensation solution

7.5.2 Photometric measurements

Adjust the apparatus to zero absorbance with respect to water prior to each measurement. Measure the absorbance of each solution in a cell of optical path length 4 cm (see note) using the spectrometer set at the wavelength of maximum absorbance (approximately 510 nm), or the photometer fitted with the filters specified. The absorbance of the zero standard shall be deducted from the absorbance obtained for the other standards.

NOTE - Cells of path length 5 cm may be used if 4 cm cells are not available.

7.5.3 Plotting the calibration curve

A graph having, for example, the mass of iron contained in 100 ml of the standard colorimetric solutions, expressed in micrograms, as a basis and the corresponding values of absorbance shall be plotted as ordinates. A new calibration curve shall be prepared whenever a new stock solution is prepared.

7.6 Determination

7.6.1 Preparation of the test solution

Add the test portion to a suitable size beaker (400ml) and 25ml of hydrochloric acid solution (4.1) and add approximately 100 ml of water. Boil until the sample has completely dissolved, and then for 5 minutes. Cool to ambient temperature. Transfer the solution to a 500 ml one mark volumetric flask, dilute to the mark with water and mix well.

7.6.2 Adjustment of pH

a) selection of aliquot portions of the test solution according to the expected iron content, as indicated in Table 2.

Table 2 – Expected iron content

Expected iron content mg/kg Fe	Volume of aliquot Portion, ml
100	50
100 to 250	25
250 to 500	10
500	5

The first aliquot portion shall be added to a beaker of suitable size (100 ml), and the second to a 100 ml one-mark volumetric flask.

Shall then be added the following:

- 5 ml of hydroxylammonium chloride solution,
- 5 ml of the 1,10 phenanthroline solution,
- 25 ml of the buffer solution.

Check the pH of the solution with the pH meter or paper indicator. The value shall be between 3.4 and 4.2. If the value is not in this range, adjust the pH by adding slowly, stirring after each addition, either the necessary amount of the acetic acid solution or the necessary amount of the sodium acetate solution. Record the amount of reagent needed to adjust the pH and reject the solution.

b) Formation of the coloured complex

Add the same amounts of all the reagents used for the preliminary test to the aliquot portion in the volumetric flask. Dilute to 100 ml, mixed and allow to stand for 10 min.

c) Photometric measurements

Adjust the apparatus to zero absorbance with respect to water prior to each measurement. Measure the absorbance of the test solution and the blank solution in a cell of optical path length 4 cm, using the spectrophotometer set at wavelength of maximum absorbance (approximately 510 nm), or the photometer fitted with the filters described in section 4. The absorbance obtained for the sample.

NOTE - If 5 cm cells were used to prepare the calibration graph, use 5 cm cells here also.

8. Expression of results

Using the calibration curve, the mass, in micrograms, of iron in the test solution and in bulk solution shall be determined corresponding to the absorbance of the zero standard.

The iron content, expressed as milligrams of per kilogram on moisture free basis, shall be calculated as follows:

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$$\frac{m_1 - m_2}{m_o} X \frac{500}{V} X \frac{100}{(100 - M)}$$

Where,

m_0 is the mass of the test portion;

m_1 is the mass of iron found in test solution;

m_2 is the mass of iron found in blank test solution;

V is the volume of the aliquot portion of the test solution (in ml);

M is the moisture content determined in accordance with ES ISO 2483.

9. Test report

The test report shall include the following information,

a) a reference to this Ethiopian Standard;

b) the result and method of expression used;

c) all information necessary for complete identification of sample;

d) any unusual features noted during the determination; and

e) any operation not included in this Ethiopian Standard or in the Ethiopian Standard to which reference is made, or regarded as optional.

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Ethiopian Standards Agency

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Organization and Objectives

The Ethiopian Standards Agency (ESA) is the national standards body of Ethiopia established in 2010 based on regulation No. 193/2010.ESA is established due to the restructuring of Quality and Standards Authority of Ethiopia (QSAE) which was established in 1970.

ESA's objectives are:-

- Develop Ethiopian standards and establish a system that enable to check weather goods and services are in compliance with the required standards,
- Facilitate the country's technology transfer through the use of standards,
- Develop national standards for local products and services so as to make them competitive in the international market.

Ethiopian Standards

The Ethiopian Standards are developed by national technical committees which are composed of different stakeholders consisting of educational Institutions, research institutes, government organizations, certification, inspection, and testing organizations, regulatory bodies, consumer association etc. The requirements and/ or recommendations contained in Ethiopian Standards are consensus based that reflects the interest of the TC representatives and also of comments received from the public and other sources. Ethiopian Standards are approved by the National Standardization Council and are kept under continuous review after publication and updated regularly to take account of latest scientific and technological changes.
Orders for all Ethiopian Standards, International Standard and ASTM standards, including electronic versions, should be addressed to the Documentation and Publication Team at the Head office and Branch (Liaisons) offices. A catalogue of Ethiopian Standards is also available freely and can be accessed in from our website.

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International Involvement
ESA, representing Ethiopia, is a member of the International Organization for Standardization (ISO), and Codex Alimentarius Commission (CODEX). It also maintains close working relations with the international Electro-technical Commission (IEC) and American Society for Testing and Materials (ASTM).It is a founding member of the African Regional Organization for standardization (ARSO).

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